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by Members of the Graduate Program, Southern Illinois University/Carbondale: Marvin Jensen, Phil Baldwin, Steve Brunst, Laurie van Houten, William Ard, Janice Nathen, Randy Jones, Professor L. Brent Kington, and Professor Richard Mawdsley

Compiled and Edited by William Ard

February 1979

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INTRODUCTION—Unsuccessful attempts by modern technology to duplicate ancient metalworking techniques may have failed partly because they ignored the technology of the men who invented them. Neither the Etruscans nor the ancient Japanese had modern kilns or chemistry, but they did have a firm grasp of granulation, solderless joints and mokume-gane.

Interest in blacksmithing at Southern Illinois University has led to an exploration of forge joining techniques. As the coal forge or charcoal brazure was the only heat source available to the ancient smith, we consider that it created a distinct and possibly beneficial environment. This has led us to explore the use of the reduction environment as a tool.

Marvin Jensen, mokume vase form of copper, shakudo and shibuichi

BACKGROUND—SIU experimentation into forge welding began in 1969 with the development of a pattern-welded method of making "Damascus" steel. The research team at that time (no. 1) evolved a method of making patterned steel by forge welding alternating layers of steels having varying carbon, sulphur and nickel contents.

Additional pattern was developed by the mechanical processes of material removal by chiselling or grinding, surface distortion by hammering, or by twisting; subsequently forged or ground flat, the finished billet had a cuttle-bone or wood grain texture that could be brought out with a light etch.

These methods and their results have been documented in some detail in "Decorative and Sculptural Ironwork" by Meilach. (no. 2)

Phil Baldwin began using a traditional Japanese method for patterned steel in 1974. As stated in Robinson (no. 3) the process consists of the forge welding of two layers of different steels. This billet is then folded and rewelded some 15 to 20 times resulting in over 1,000 layers of steel, with carbon repeatedly being introduced by putting straw ash in the welds.

A fine, delicate pattern is developed by the folding process, but additional pattern can be developed by exposing the stratification by the means mentioned above, or by simply forging the billet at an angle and exposing the edge of the layered stack in a wood grain effect.

At this point investigation began into the bonding of non-ferrous metals. Early readings in C.S. Smith's "History of Metallography" (no. 4) led to capillary quench diffusion bonding. This method of quenching rivetted layers of various alloys into molten metal gave a bond that was only occasionally successful. Done properly, the process resulted in a strong, malleable bond, but was abandoned due to superior results in forge welding.

Early Test Piece: mokume showing pattern developed from behind by dapping and filing.

Sheffield plating (or "silver cladding") (no. 5) was also looked into at this time, but though successful with silver, copper and copper alloys, it was less efficient than forge welding and set aside for future study. Experimentation here led us to drop the use of zinc alloys due to bond strength problems and violent silver-zinc reactions problems.

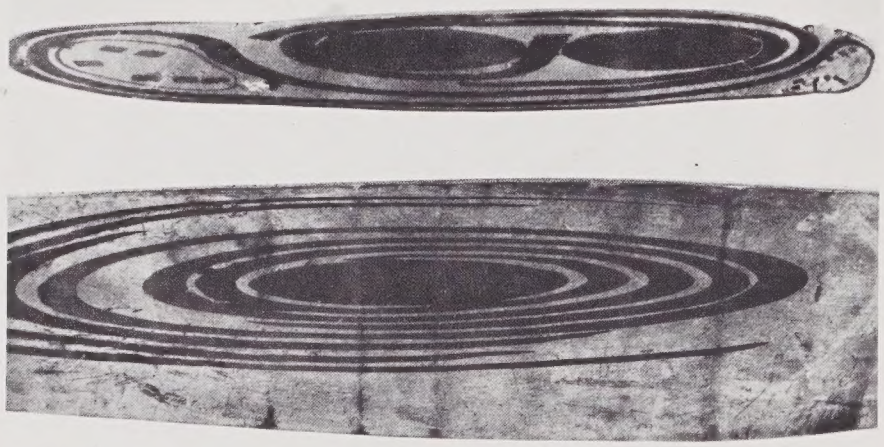
An article in a 1914 "American Blacksmith" (no. 6) taught us to forge weld copper and copper alloys successfully and turned our attention to traditional mokume-gane as a cleaner, more controllable bonding method.

Research into Japanese methods was indicated, and a 1977 exchange workshop with Gene and Hiroko Pijanowski provided much needed information on traditional copper alloys. The workshop also introduced new information on a chemical patina (Rokusho) used to differentiate the alloys in the finished piece.

One mutually beneficial discovery was the superiority of the coal-fired forge over the electric kiln in producing consistently good results; credit was given the non-oxidizing environment of the coal fire. With the new alloys we were achieving close to 100% success rate.

The final development was the introduction of the torque-plate in 1978 by Marvin Jensen. By clamping the layered stack between two steel plates, not only was our metal preparation time cut by 2/3 and a good, non-slip contact between layers insured, but properties of the metals themselves were used to insure a good bond. The difference in the

1. Daryl Meier, Robert Griffith, and Jim Wallace.
2. Decorative and Sculptural Ironwork; Meilach; pp. 216-232.
3. Art of the Japanese Sword; Robinson; pp. 25-26.
4. History of Metallography; Smith.
5. History of Old Sheffield Plate; Bradbury.
6. Forge Welding Copper and How It's Done; Constantine; p. 291.



Early Test Piece: rolled capillary quench billet.

expansion coefficients of copper and steel insure that the copper alloy layers will expand faster than the steel clamps, driving out the oxygen between the layers and seating the alloys tightly against each other. (no. 7)

7. Metals Reference Book VII; Smithells; pp. 642, 346.

Steve Brunst: pattern development experiment.

MOKUME—Early attempts to copy the mokume process included Prof. Roberts-Austin's turn-of-the-century "imitation" mokume (no. 8) where the layers of metal are hard soldered together.

A Japanese variation mentioned in Hiorns (no. 9) involved pouring semi-solidified alloys together in a box. Called kasumi-uchi (no. 10), this produced a mottled, "marble-cake" billet and a pattern is developed in ways similar to mokume.

The actual mokume-gane process, about 300 years old (no. 11), is the diffusion lamination of copper and the copper alloys shibuichi, shakudo, and kuromi-do. Silver and gold are also used. The alloys are rolled out, cut into sheets and stacked in a contrasting sequence. Traditionally the stack is placed in a steel box or bound with heavy wire with a steel plate on top to provide weight (no. 12), and then fired in a charcoal oven.

Diffusion takes place as the molecular movement within the crystalline structure of the metal is increased with the introduction of heat. This encourages the crystal lattices of the adjacent, mutually soluable metals to grow into each other across the weld boundary.

The diffusion bond is characterized by the welding of the parent metals at a temperature significantly lower than their melting points. In some cases, as with copper and silver, a eutectic alloy is formed between the two metals and the necessary welding temperature drops even lower.

The Pijanowskis used an electric kiln in the interest of scientific control and measurement, but we felt a return to the forge environment would provide an atmosphere less conducive to oxidation (no. 13).

8. Mixed Metals; Hiorns; p. 274.
9. Ibid; p. 275.
10. Workshop: Mokume-gane; Pijanowski (Craft Horizons).
11. Ibid.
12. Laminations of Non-Ferrous Metals by Diffusion; Pijanowski (Goldsmiths Journal).

13. Research team of 1977: Marvin Jensen, Phil Baldwin, Kathy Doyle, Steve Brunst, William Leth, Steve Miller, under direction of Prof. L. Brent Kington.

THE PROCESS: ALLOY TO BILLET—The first of the mokume alloys manufactured at SIU was shakudo. First found in Japan in a sword of the Mitsutada school, the alloy was used extensively along with bronze in sword furniture of that era (1335-1357 A.D.). (no. 14) It consists of a formula of copper (94-96%), silver (1½% or less), and gold (4-5%). The presence of not less than 4% gold is essential for best coloring (no. 15). Shakudo is a dark copper color and takes on a purple-black patina in Rokusho.

Shibuichi was next with a formula of copper (51-68%), silver (32-49%), and 1/10% or less of gold. First seen in sword furniture of the Ashikaga Period (1338-1573), it came in 15 different grades. The most popular grade was a formula called "sambo-gin" consisting of 67% copper, 32% silver and a trace of gold (no. 16).(Shibuichi with a higher silver content was found to have too

14. Metals and Metalworking in Old Japan; Gowland, pp. 90-94.
15. Mixed Metals; Hiorns; p. 274.
16. Metals and Metalworking in Old Japan; Gowland; pp. 90-94.

Marvin Jensen: Bank with mokume and silver construction.

low a melting point to be used safely in mokume laminates. Our alloy of 5% silver or less will not melt in the bonding process and colors, upon patination, very much like traditional Shibuichi.) It is a pale bronze color and a rich gray when patinaed.

Last is Kuromi-do, a formula of copper and 1% arsenic (no. 17). While it is not safely manufacturable in the studio, the commercial alloy is safe to use in the mokume process. It possesses a dull bronze color with a brown to blue-black patina.

Any variations of copper, gold or silver content are likely to produce alloys suitable for bonding to copper and to themselves. Pure copper, gold and silver can also be used to provide further contrast in layering.

Brass can also be included in the stack if it is kept separated from the silver alloys. Adequate bond strength can be achieved with brass, but the resulting product does not have the malleability of copper-silver-gold systems and care must be taken not to overwork the brass elements in the laminate.

Mix the alloys by weight in a clean graphite crucible. We use fine silver, fine gold, and salvage electrical copper because of its availability and its low sulphur and oxygen content. The fluxes used are sal ammoniac (ammonium chloride), against copper and zinc oxides, and borax to collect other impurities into the slag.

As the crucible is raised to melting temperature in the forge, add a little borax (do not overflux) and either charcoal or wood chips. This forms a layer of carbon on top of the alloy and prevents it from oxidizing. Great care should be taken to avoid oxygen contamination because it will cause shortness; if the product is short (i.e. brittle) it will have to be remelted.

A forge note: once the crucible is in place, it is important to keep pushing coke into the firebox and down around the crucible. If the fire is allowed to hollow out, the intense melting temperature will be lost.

When the alloy is completely liquid, stir it with a carbon rod and remove it from the forge. Do not pour the alloy too hot because crazing, an effect of a too-large crystal structure, can result if the metal is cooled rapidly from this state.

When the metal has cooled slightly, throw in more wood chips and pour while they're still flaming. This provides a strong reducing atmosphere in the mouth of the crucible to further protect the alloy while pouring.

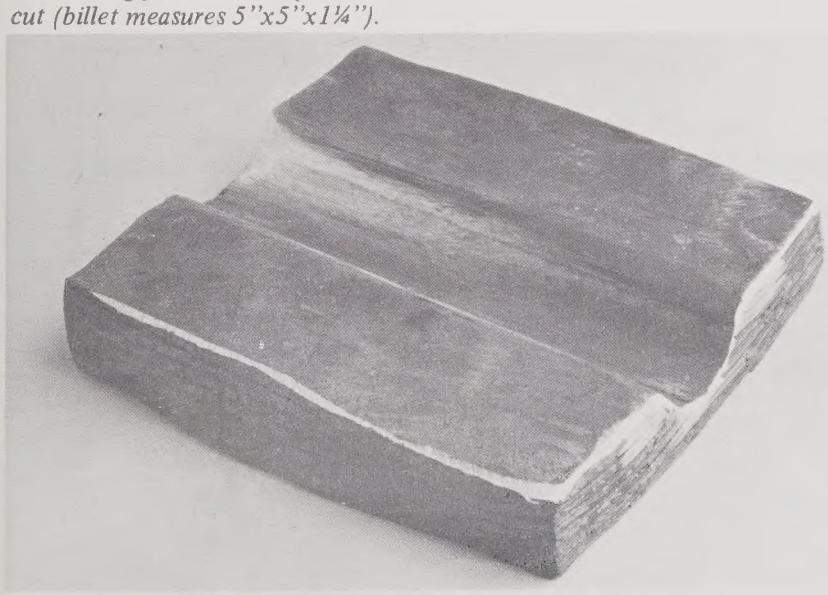
The ingot molds should be coated with acetylene soot and heated to around 400 degrees F. This is the temperature at which paper chars and is hot enough to keep the metal from freezing too quickly. Pour slowly and evenly down the side of the mold to prevent splashing and inclusions. There is no need to skim the carbon from the metal.

Hot forge the ingot lightly to align the crystal structure and improve plasticity. We suggest a plasticity test on the quenched ingot at this point: clamp it in a vise and bend. A fracture indicates shortness from oxygen or other impurities and the ingot should be remelted.

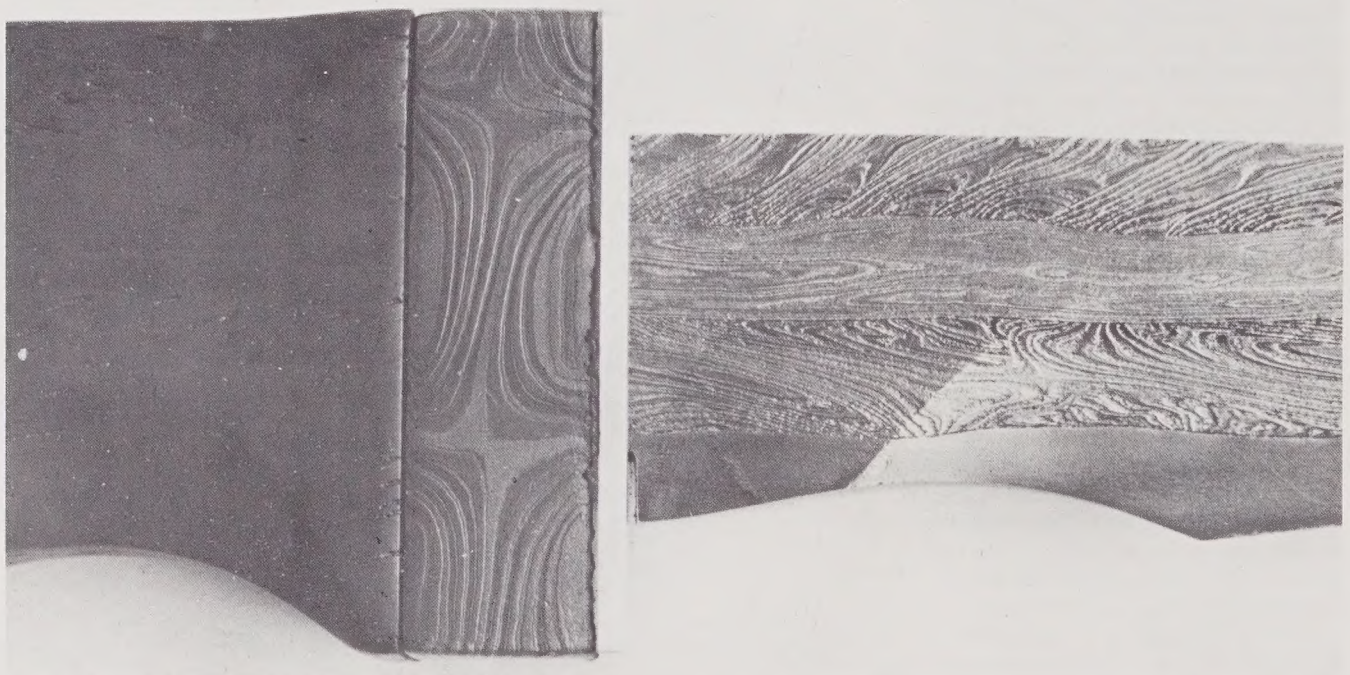
17. Laminations of Non-Ferrous Metals by Diffusion; Pijanowski.

Marvin Jensen: Bank (copper, shibuichi, kuromi-do) raised from end slice of 33 layer billet, 2½" dia.

Forged mokume billet of 32 layers showing first rounded pattern cut (billet measures 5"x5"x1¼").



Phillip Baldwin: Man's Bracelet in silver-copper mokume; pattern developed by twisting and punching.



Phillip Baldwin: closeup of Fuchi twist pattern mokume knife fittings on a twist section Damascus blade.

Otherwise, roll the ingot out and cut it into sheets. Make sure all the layers are reasonably clean and flat; minor warpage and scratches are permissible, but oxides and oils should be removed by pickling and a light pumice scrub.

Then stack in any sequence, usually at least eight layers, bearing in mind contrast and the possible migration of the arsenic in the kuromido, which could darken the color of the layer next to it. The bottom layer, to make the most of the pattern possibilities of the laminates, should be a thick sheet of copper. We use a 3/8 inch electrical bus bar, a "clean" alloy available in heavy plates. This layer will form the inside of the finished piece and allow the use of all the laminated layers in pattern development.

Clamp the layered stack in the torque plate. For the construction of the torque plate, use a ¼ inch to ½ inch steel plate, top and bottom, depending on the size of the billet, and six to eight 3/8 inch bolts. Mild steel works very well, though an "s" type or stainless steel would last longer and oxidize less. Some sort of parting medium such as ochre should be put between the copper and the steel to prevent fusion, and the bolts tightened to around 50 ft. lbs. for a large billet. This insures good contact between the sheets and guarantees no slippage. It also sets the stage for expansion pressure to help insure a clean bond.

The heat for a large billet should be done in a hooded fire using "blacksmiths" or "metallurgical" coal, which is high in BTU output and low in sulphur and ash. A hooded fire is one which is roofed over with dampened coal ("green coal") like an igloo with a firebrick arch in front to receive the billet.

This type of fire is built by pushing dry coke over the fire, making a small mound, and then covering the mound with green coal. Fed with air from the tuyere below, the mound will quickly hollow out, leaving a coke walled oven filled mostly with gas from the fire underneath and very little oxygen. Finally, slide steel flatstock across the fire to support the billet and push the billet into the fire.

A hooded fire is recommended, particularly for those unfamiliar with a coal forge, but a trench fire can also be used with the torque plate. With this method a handle is welded to the torque plate and the billet is placed directly into the fire, buried in the coals. The trench fire is easier to make, but it has two drawbacks: it is difficult to see the billet and check on the progress of the diffusion, and it is very easy for the uninitiated to melt the billet.

Taking care not to let the fire die down, rotate the billet in the hooded fire to avoid burning the back corners. The steel plates will prevent this to some extent, but the side away from the doorway tends to overheat.

At some point after the billet has achieved an even, red-orange glow, a "shadow" will darken the whole billet for a moment. This is a sign of a rapid drop in temperature or other molecular activity; it is, at any rate, a cue that the diffusion is almost complete.

The diffusion of copper alloys will occur at approximately 2/3 the melting termperature of the metals, happening gradually between 1400 and 1900 degrees F. Copper and silver, having a high degree of mutual soluability, will bond at 2/3 the temperature of the alloy that forms between them, between 1100 and 1400 degrees F.

In an article in the Goldsmiths Journal (no. 18) John McClosky mentions a way to calculate the precise

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18. A Metallurgical Analysis of the Lamination of Non-Ferrous Metals; McClosky (Goldsmiths Journal No. 16).

Bond strength tests on fine silver. From left to right: CuChloride and Antimony 03 80/20, CuChloride and Antimony 03 50/50, Antimony 03 alone.

time and temperature to fire any particular billet. For our purposes, however, it is much easier to watch the visual indicators of the metals themselves and take the billet out when it is done.

When the shadow has passed and the temperature builds back to where it was, reach in and scratch the joints and across the layers with a stainless steel probe. A "silvering" or a mercury-like shine in the scratch indicates the diffusion has taken place. Remove the billet and, placing it on the anvil, strike it lightly and rapidly, working around the top of the steel plate.

Take the torque plate off, cut it off if necessary, and return the billet to the fire. Forging improves the quality of the joint, so we generally work the billet down to 50-60% of its original thickness immediately. Work from a cherry red heat into black; a higher heat won't hurt the bond, but it will promote arsenic migration if kuromi-do was used.

While this may seem a complicated procedure for the average jeweler in his studio, we should point out that although our largest billet was a four inch cube of 32 layers weighing 16 pounds, the smallest was a ¾"x5/8" billet of 8 layers and was fired in a trench fire in 15 minutes.

Using the torque plate, excellent results could be achieved in an electric kiln as well, with a localized reduction environment provided by painting hide glue on the sides of the billet. This would quickly turn to a layer of carbon and help prevent oxygen contamination.

With the invention of the torque plate, bonding success was taken for granted and we turned our attention to the inconvenience of the hooded fire. In addition to the danger of overheating the back corners, there was the danger of damaging the hood in rotating the billet, as well as the inconvenience of disturbing the billet with constant stoking.

This led to the development of the forge/kiln which we use today (see diagram). Here the heating is direct but separate, the kiln being well above the firebox. This arrangement heats more evenly, keeps the stoking separate and the peep-windows provide a 360 degree view.

Steve Brunst: Container (copper, shakudo, shibuichi, kuromi-do) raised from 55 layers.

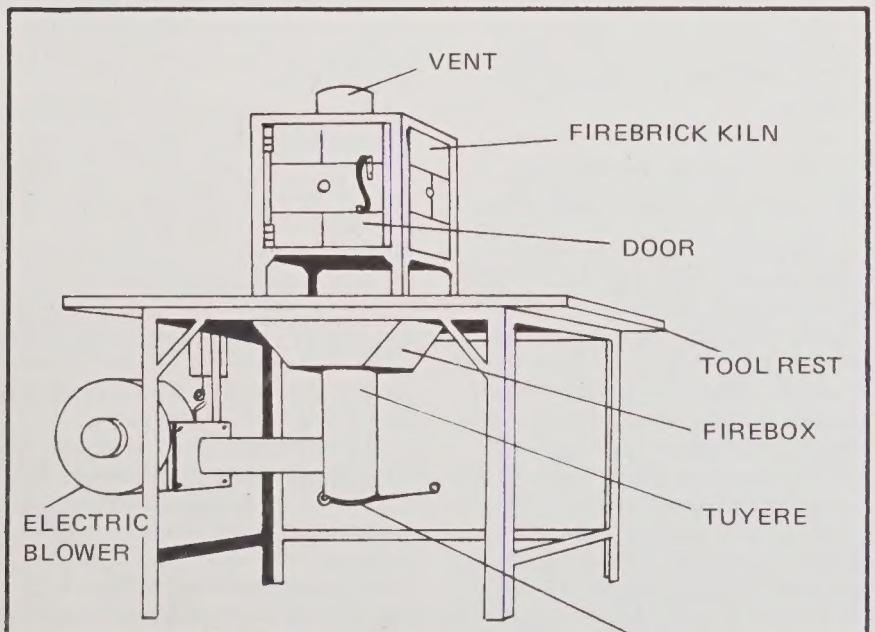


FIGURE A: THE MOKUME FORGE/KILN
–Height (total) 53", width 42", depth 28"
–Height (kiln) 22", width 16", depth 14" (outside)
–265 CFM Dayton Blower

THE PROCESS: BILLET TO SHEET (Pattern Development)—The pattern development techniques for mokume are similar to those used on Damascus steel, namely material removal, surface distortion, edge forging, and twisting. The philosophy is much the same as well, the object being the creation of a delicate surface pattern requiring an involvement on the part of the viewer deeper than mere attention to form.

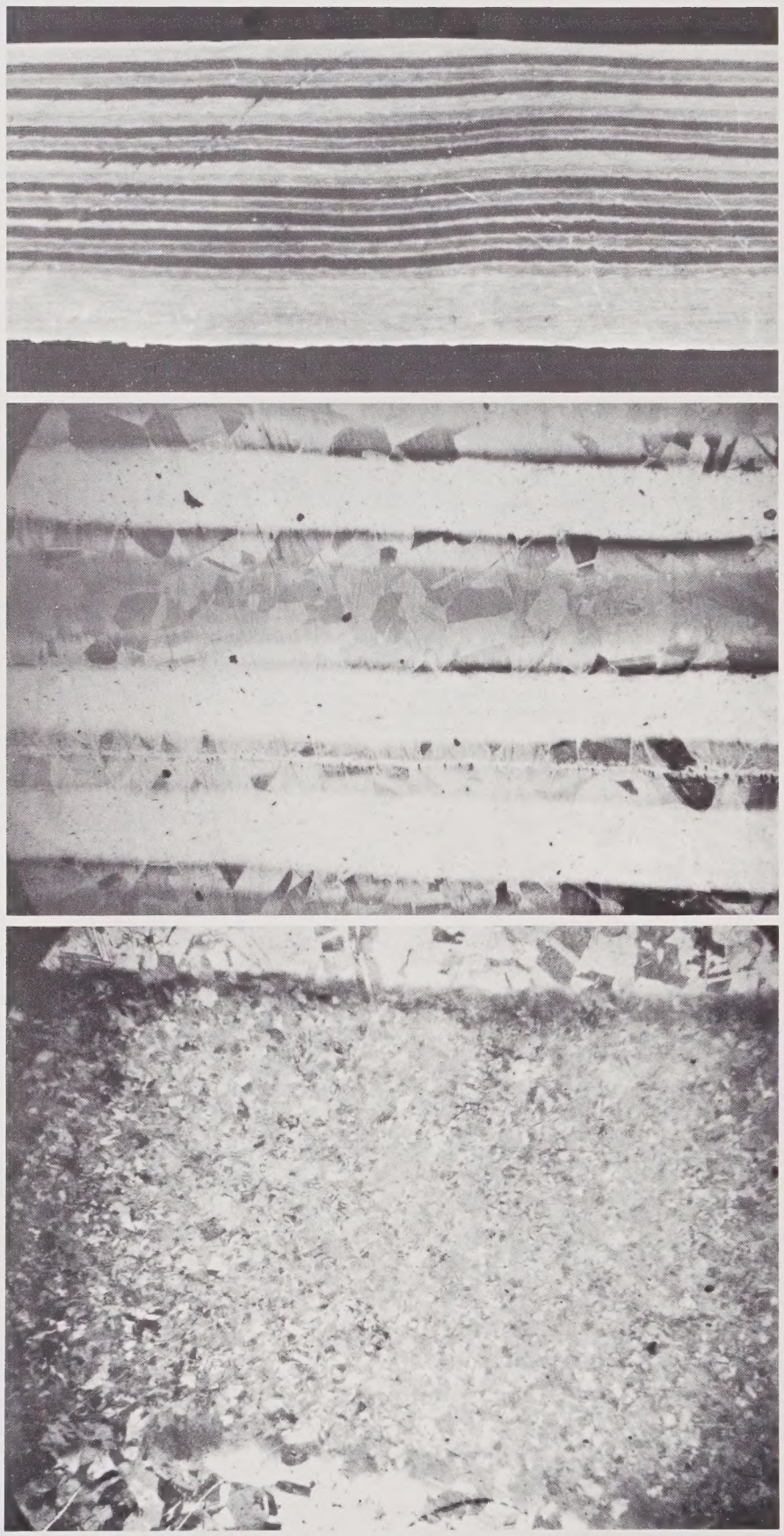
A mokume pattern produces a "visual sensation combining that of a resolvable abstract design with that of pure texture" (no. 19). The woodgrain effect of the pattern is a form of artificial identity with nature, and its look of a topographic map provides an artificial depth, an "interplay of dimensionalities" that is at once subtle and absorbing.

The mechanical means of achieving these patterns are somewhat less subtle. Development by material removal is usually done with a hammer and chisel. By carving the billet with a chisel, great control can be exercised at the expense of hours of work. The Pijanowskis suggest the use of a two-flute ball end milling attachment in a vertical mill to produce a cut like the hatsuri-tagane, the traditional chisel (no. 20). This tool can also be used in the drill press to some extent, drilling more even designs than a drill bit.

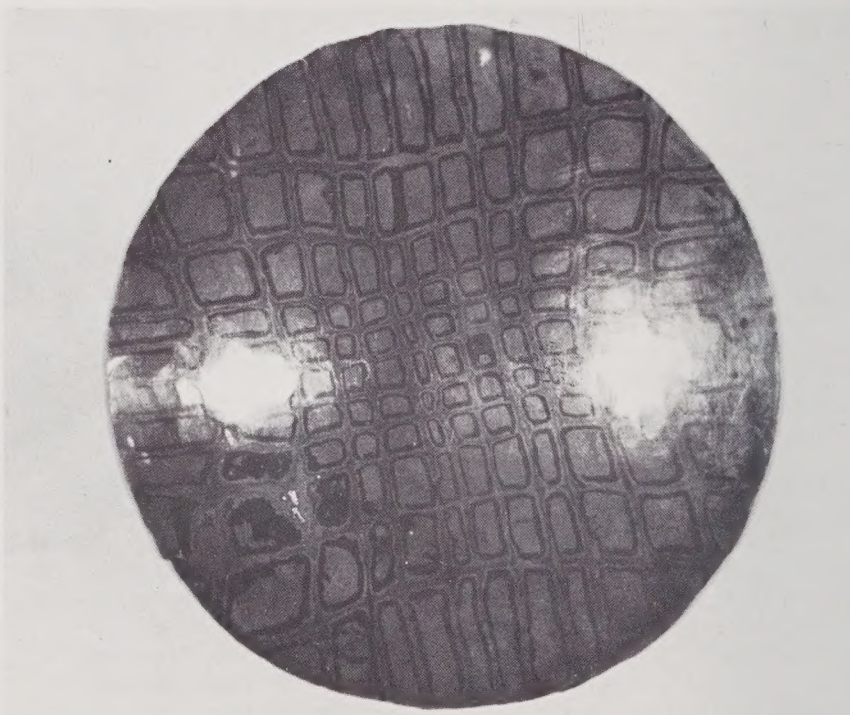
Experimentation has also been successful with turning concentric

19. Sectioned Textures in the Decorative Arts; Smith.
20. Lamination of Non-Ferrous Metals by Diffusion; Pijanowski.

FIGURE B: Photo-micrographs of the metallic grain structure characterizing a diffusion bond. Note the actual growth of crystals across the weld boundary. (photos courtesy Brookhaven National Laboratories.)



Steve Brunst: grid pattern mokume disk.



rings of varying depth on a machine lathe.

The surface distortion methods consist largely of dapping patterns into the billet from the back after forging and rolling it to a suitable thickness. These methods produce much softer, more rounded patterns when the resulting surface bumps are filed flat.

A Damascus twist pattern was also found successful, producing a subtle rope-like pattern when the billet is forged and ground flat.

The most unusual design experiment involved making mokume from mokume. Here, end slices were cut from an unforged billet and stacked with the laminates turned in different directions. This composite billet was bonded, forged and turned on a lathe resulting in an abstract pattern with a linearity unlike traditional mokume.

Before starting the pattern development, it is a good idea to cut off the edges of the billet. This will discourage stress risers from the uneven joints in the later forging process, and it affords a chance to check the bond before investing time in the pattern.

The pattern cuts should be smooth and rounded; deep, narrow cuts will close in with forging and do not use the stratification to its best advantage in any case.

If fine lines are desired, they can be developed in multiple pattern development stages. This method is to go back and recut parts of the pattern after forging when a narrow cut can be made without so much chance of forging it shut.

The finished product should have the same workability as copper sheet. The copper alloys used have the same malleability and, if no zinc or tin alloy is used, the same tenacity. If bonded properly, it will handle the same as copper; it will raise, forge and draw well with no fear of delamination, and can be annealed frequently without problems. Richard Mawdsley has succeeded in drawing mokume into tubing with no difficulty.

The manufacture of mokume sheet is no longer a technical problem. The artist's real effort should be invested in aesthetic development through an examination of the relationship between pattern and form. There is a tendency, after all this time and effort, to think of the material itself as "art". This is probably the reason for so many boring mokume-gane pieces and such a tendency should be watched carefully.

PATINA—During the workshop with the Pijanowskis we learned of Rokusho, the patina which lends such a rich variation of color to mokume. A sample was analyzed by chemists at both Purdue and SIU and the resulting reconstruction was workable but not entirely satisfactory.

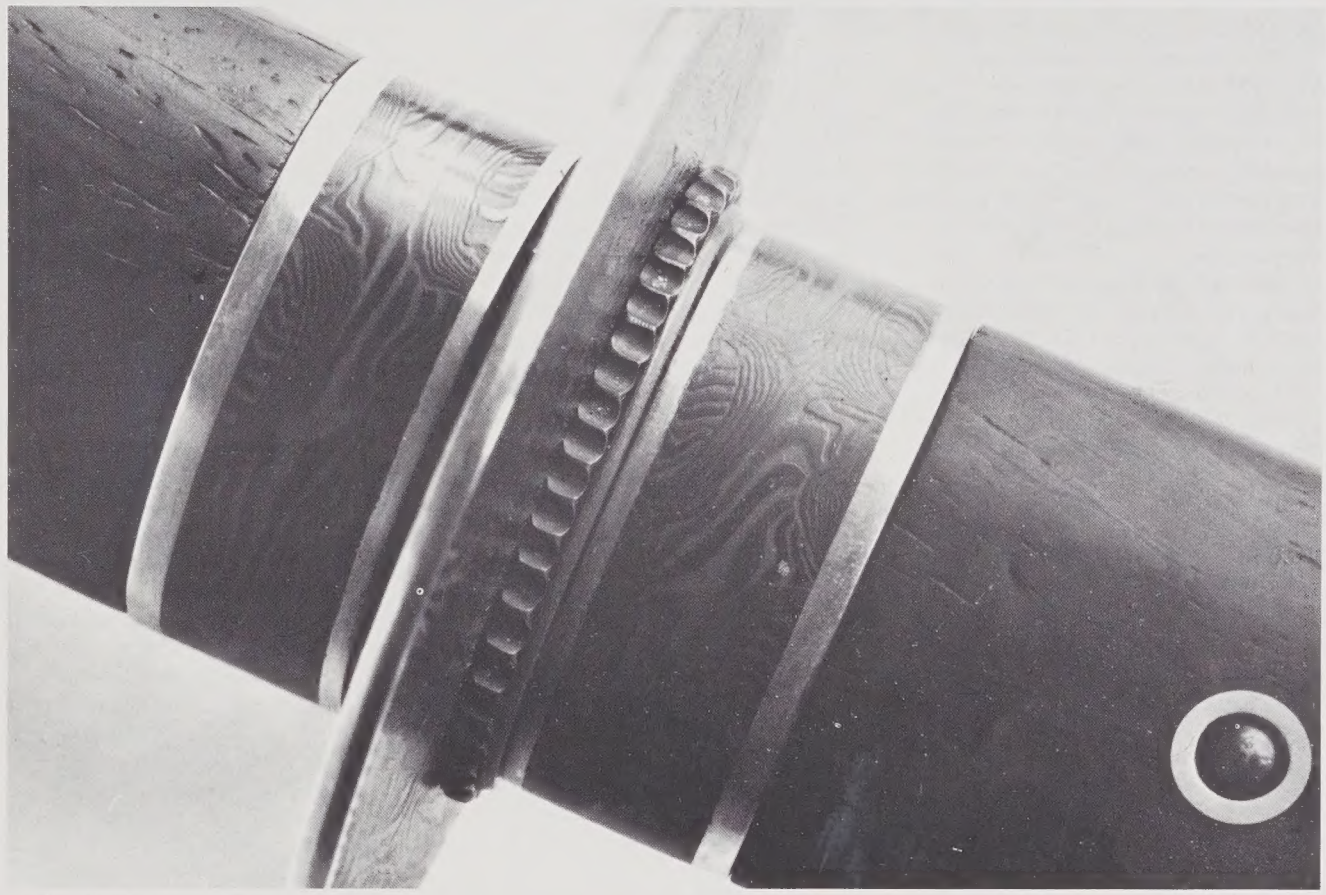
Research into Chinese and Japanese patinas turned up repeatedly mention of vinegar of plums and verdigris as main constituents (no. 21). Eventually we were able to translate the active ingredient in verdigris as cupric acetate and we had a formula going.

Phil Baldwin discovered a mention of the importance of salt in Robinson's "Art of the Japanese Sword" under the "care and cleaning of shakudo sword guards" (no. 22). Experimentation yielded a recipe that gives colors close to traditional models: 4 dwt (6 grams) cupric acetate; 1 dwt (1.5 gr.) copper sulphate; 1 dwt (1.5 gr.) uniodized table salt; 1 litre distilled water.

Although the color quality was very good, it was being marred by spotty coverage until Baldwin happened on an answer to the problem. In the slightly acidic Rokusho, introduction of a copper bar in the solution with the mokume achieved consistently good results by a galvanic response. The nature of the action is still unsure, but the results proved reliable.

In retrospect, it seems probable that the ancient smiths avoided the problem, boiling their patinas in a copper pot instead of a pyrex one.

The patina procedure is fairly simple. Texts indicate boiling the piece first in lye prepared by lixiviating wood ashes (no. 23), but we found a light nitric etch works well also, and the Pijanowskis recommend cleaning in potassium cyanide (no. 24).



Phillip Baldwin: closeup of sterling-copper mokume sword furniture (pattern developed by twisting and splitting a cross-sectioned billet; etched in 25% nitric and colored in our Rokusho).

Before coloring, the piece should be cleaned with pumice to remove surface oxides and oils to improve the evenness of the patina. If the piece is buffed, take care to remove all traces of wax or oil.

Boil the piece in the Rokusho until the desired colors are achieved. This takes about 15 minutes in our recipe. The colors can be varied according to the amount of salt used; copper, for instance, can be colored from almost a purple to a leathery orange-brown.

The finished piece should be washed thoroughly and coated with wax by heating the piece and applying the wax by hand. This not only protects the color of the patina, but it imparts a soft shine to the work even if it was only given a pumice finish before coloring.

The pattern can be dramatically heightened by the use of an electro-etch before, or instead of a patina. This would give actual relief to the surface of the work and serve to preserve the pattern on a piece of jewelry exposed to wear and tear where the patina is likely to wear off. It can also serve to lend some life to an otherwise uninspired pattern.

This concluded our research into mokume-gane. We feel our work has culminated in a process by which an artist, with some experience, can manufacture mokume sheet in quantity without a great deal of trouble, freeing him to use mokume-gane as he would any precious metal.

21. Art of the Japanese Sword; Robinson; p. 94; Mixed Metals; Hirons; p. 274.
22. Art of the Japanese Sword; Robinson; pp. 94-95.
23. Science and Civilization in China; Needham; pp. 250-255; Art of the Japanese Sword; Robinson; p. 95; Metal and Metalworking in Old Japan; Gowland.
24. Lamination of Non-Ferrous Metals by Diffusion; Pijanowski.

INTRODUCTION TO GRANULATION—Our success at mastering mokume in the forge led us to investigate another ancient metal joining technique: granulation. The two processes seemed related not only by the type of technology which fostered them, but by their metallurgical principles as well.

Whereas the diffusion bond for mokume is the migration of crystalline structure across a weld boundary, granulation employs the actual fusion of the surfaces to be joined. Fusion, as it applies to granulation, can be described as the use of a metal or metallic oxide to create an alloy with the parent metal at the contact point of the surfaces being joined. Fusion occurs at the melting point of the alloy so that a minimum application of heat is necessary.

An eutectic is an alloy of two or more metals combined in a ratio achieving the lowest possible melting point in the system, and having the property of melting immediately into a liquid phase. Eutectics are ideal alloys for granulation, but not all the alloys used are eutectics. An alloy for granulation should be able to melt at a relatively low temperature and diffuse rapidly into the parent metal.

BACKGROUND—Granulation can be defined as localized fusion welding, using the on-site formation of an alloy instead of intense localized heat as in arc welding. Davidson (no. 1) illustrates the desirability of localized fusion over autogenous welding or sweating, where the whole surface is fused (no. 2), and over soldering, where there is no true molecular bond. The true granulation process, she felt, was a type of brazing most closely related to Littledale's colloid hard soldering.

Our first clear understanding of the process came from Littledale's patent papers (no. 3) and a transcription of one of his lectures on the subject (no. 4). Here he pointed out the drawbacks of hard soldering and explained the copper-silver and copper-gold eutectic systems. His process was to put his work together with a dilute solution of copper salt (cupric hydrate) and "Seccotine", a commercial hide glue. Under heat, the hydrate converts to a copper oxide and the glue begins to carbonize. The oxygen in the oxide then combines with the carbon and pure copper is reduced from the oxide around 890 degrees C. The resulting streak of copper in the joint, at that temperature, unites with the parent metal and "alloys" the joint closed.

Our earlier readings had suggested a possible connection between copper residues and the solderless joining of silver and gold. Recipes from the Mappae Clavicula, a medieval document, include gold solders made from copper oxides (no. 5), and Benvenuto Cellini tells of soldering on a charcoal bed with verdigris and borax (no. 6). Theophilus, in his treatise, gives a formula for gold solder from copper oxide and salt that will allow assembling a chalice in the forge without conventional solder (no. 7), and Pliny (no. 8) relates that "gold is dug out of the earth and in proximity to it, gold solder." Davidson translates this to mean verdigris and malachite, which are basically copper acetate and copper carbonate (no. 9).

Clearly ancient granulation formulas depended upon a eutectic alloy from copper and conditions that encourage its formation. Littledale stated that a reducing atmosphere, though not essential, improved the joints in his experiments. We were inclined to agree after our work with mokume. We began our testing with a long reducing flame from a gas-air torch and eventually built a compressed air-fed charcoal brazier for the studio (no. 10).

THE PROCESS—The actual routine of granulation is very simple, from preparing the granules to firing, and only practice makes a master.

The granules are made by the familiar method of placing wire clippings or paillons into charcoal in a crucible and melting them into spheres. Our method differs only in that the paillons are packed in wet charcoal paste and the crucible is fired in the forge. The crucible is raised to a white heat on the outside and held there for 15 minutes, then air cooled. The granules are then sorted or sifted according to size.

For the purpose of doing innumerable tests during our research, the method of torching the paillons was used to create small quantities of granules quickly. The paillons were melted on a charcoal block and allowed to roll off into water as soon as surface tension pulled them into spheres. This produces slightly elliptical granules.

Selection of a chemical will be discussed later, but generally a formula with a low firing temperature, rapid diffusability and clear visual fusion indicator is desired. The cement for our formulas was hide glue from Sears.

Place the granules or filligree with a wet watercolor brush using the surface tension of the solution to hold the design together. Use only enough solution to hold the granules in place and surround the joint. Dry the piece thoroughly.

Fire in a muffle, brazier or reduction flame; the flame was found to be the most expedient for testing. Usually, the stages that occur are these: first the glue carbonizes and the metal frosts. Then, as the piece approaches a red heat, the granules appear dark while the filletting of carbon around them is a bright red. When the solution oxidizes and combines with the carbon, the alloy is formed and the joint flushes somewhat like a solder joint. A rapid heat is essential as the carbon seems to gradually dissipate if heated for a long time.

On silver the bond appears as a mercury-like flash and a surface tension change. On gold the surface at the joint becomes liquid and crawls. The appearance of a long seam is that of a solder seam: the flash flows along it and the joint is sealed.

1. Greek Gold: Hoffman & Davidson; p. 45.
2. Greek and Roman Jewelry; Higgins; o. 36.
3. Improvements in Hard Soldering Mixtures; Littledale (Patent Specification No. 415, 181).
4. A New Process of Hard Soldering; Littledale
5. Mappae Clavicula; trans. Hawthorn & Smith.
6. Treatises of Benvenuto Cellini; Cellini; p. 46.
7. On Divers Arts; Theophilus; trans. Hawthorn & Smith.
8. via Hoffman
9. Greek Gold; Hoffman & Davidson; p. 47.
10. Research team comprised of the authors.

Early granulation test piece: 5/8" disc and grains of fine silver granulated with Antimony 03.

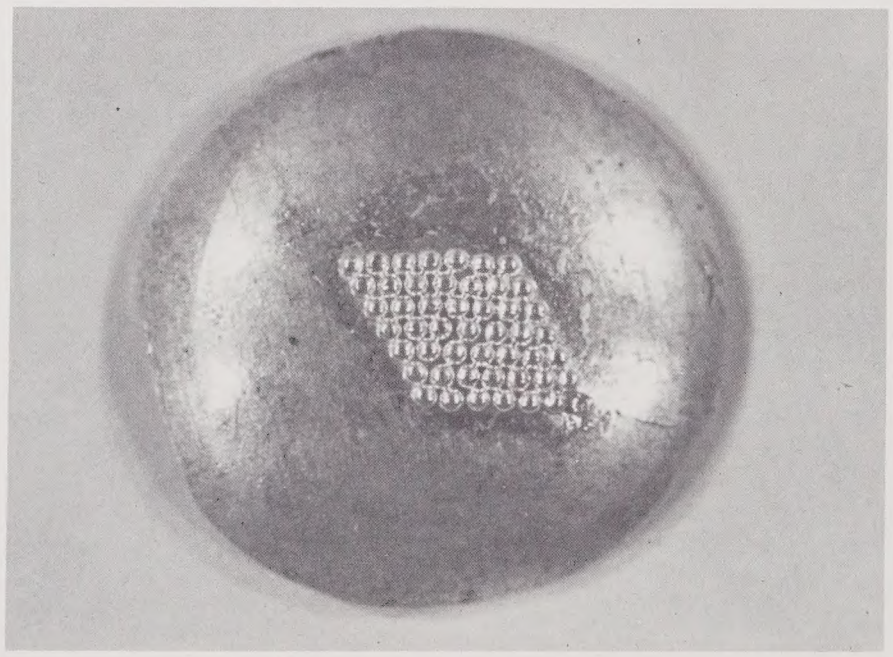


FIGURE C: Phase Diagrams Demonstrating The Characteristics Of Copper, Silver, Gold & Arsenic Under Various Alloy And Temperature Conditions (diagrams from Butts, Allison; Copper, The Science and Technology of the Metal, Its Alloys and Compounds; Reinhold Publishing Corp., New York 1954)

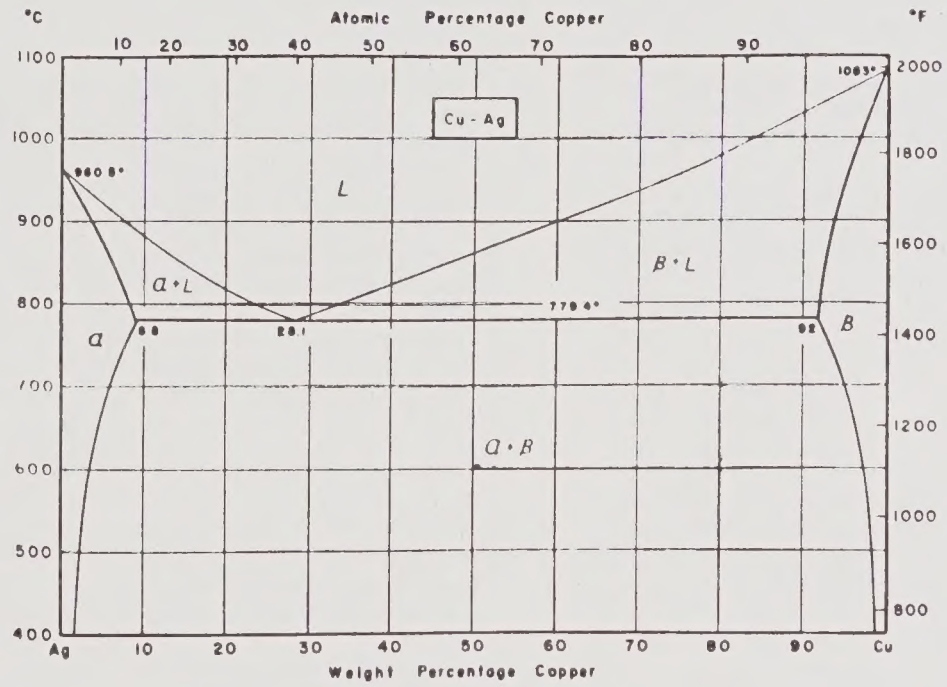


FIGURE 22-1. Diagram of the copper-silver system.

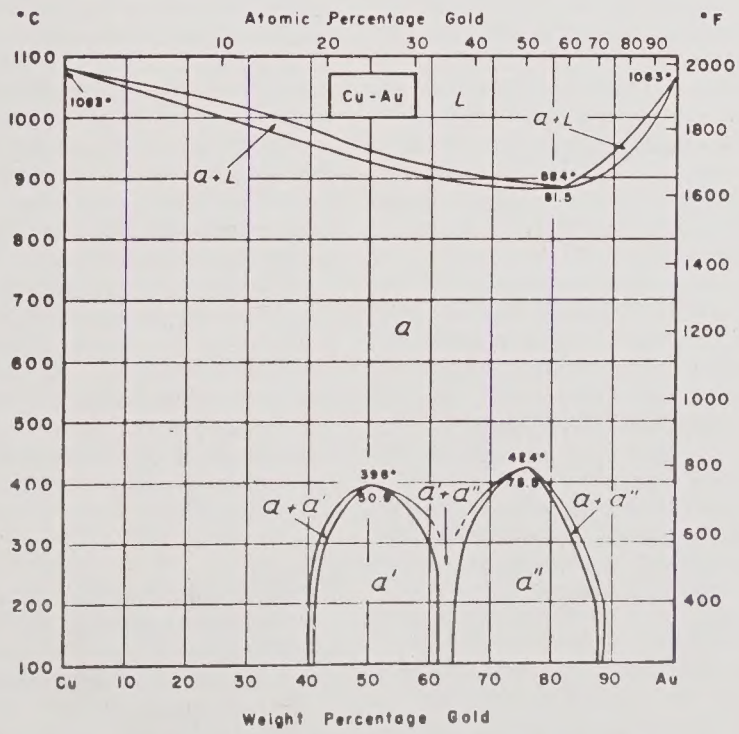


FIGURE 22-5. Diagram of the copper-gold system.

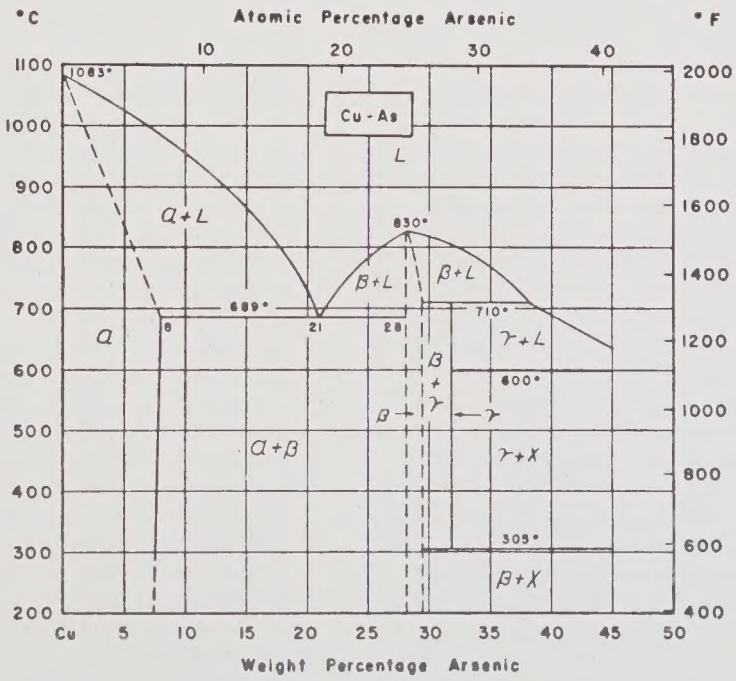


FIGURE 22-4. Diagram of the copper-arsenic system.

The appearance after firing on silver is a soft, frosted ground, shiny granules and very light fillets. Ideally, the granules should fuse only at their tangent points and appear absolutely spherical. Gold should appear very shiny and burnished both on the ground and granules, with very light fillets.

Over filletting is generally a result of overheating, though too strong a solution will also overfillet, causing pits on the surface of the piece. Too thin a solution greatly increases the firing temperature.

CHEMICAL EXPERIMENTATION— Using the principle and theory as we understood it, experimentation began on fine silver. It was understood that not all the alloys created would be eutectics, but the overriding concerns in the experiments were still low fusing point and rapid diffusion.

Beginning with Littledale's formulas and working through all of our sources, almost every formula was found to work to some extent. The most workable formulas were bracketted in dilution, generally from 30/1 to 110/1, but the least promising were abandoned after wide bracketting. The criteria for a "good" formula were good readability at the fusion point, clean surfaces without reticulation or pitting, and acceptable bond strength with small fillets. The formulas were mixed by combining the proper ratio of chemicals with a pre-mixed solution of glue and water, 3/1. Flux was added to the chemical when dry, generally at one part in four. Some of the chemicals, such as acetate, needed to be mixed into a paste before adding the glue solution so that they would go into suspension.

The chemicals listed here are all good formulas for one reason or another; some are excellent in all respects and are highly recommended. A fairly wide variety of results were achieved with each chemical through differences in application and torch manipulation, so these characteristics should be viewed as averages.

On fine silver:

1. Antimony Trioxide (Littledale) with borax glass flux 3/1 is the best overall granulation formula for fine silver. Bracketting best at 30/1, it gave good readability, very strong joints and light, regular fillets. Antimony gave unquestionably the best overall appearance with bright, shiny grains on a clean, frosted ground. Its fusing temperature was the highest of the best formulas.
2. Cu Chloride (Littledale) was recommended by John Marshall. It bracketted best at 110/1 and had very easy readability. Chloride gives a strong joint with clean, frosted grains and frosted ground; an excellent formula for strong, light fillets, fusing slightly lower than antimony 0_3 and acetate.
3. Cu Acetate (Cellini) was the third best in the group. Unlike Cellini, we found better results without using borax flux. It gave easy readability at 110/1, with a good bond, light fillets and clean, frosted appearance. Acetate was not as strong or as clean as chloride or antimony 0_3.
4. Cu Hydrate (Littledale) at 30/1 had good readability at fusion temperature and left a clean surface with shiny granules and good fillets. It did not, however, stay in suspension well and, contrary to Littledale's observations, did not seem to bubble less than the other formulas.
5. Cu Chloride-Zinc Hydrate-Tin Dioxide 50/30/20 (a variation on Littledale's "easy running" formula) using chloride instead of hydrate gave good fillets with good readability at 30/1, but bond strength and surface appearance were not as good as antimony 0_3.
6. Cu Chloride-Antimony 0_3 50/50 (Littledale variation) is an excellent formula at 30/1 giving good readability, excellent fillet structure and bright surfaces. It is not as controllable as antimony 0_3 alone: fillets build very rapidly and it is not as easily read. It fires lower than the chloride-zinc-tin formula. This formula at 80/20 left stronger, heavier fillets with the same general appearance.

Zinc Oxide and Tin Dioxide (Littledale) both gave an inconsistent fillet structure and a dull appearance. They were both abandoned due to comparatively weak bond strength.

Cu Oxide (Littledale and the Mappae) and Cu Carbonate (Hoffman) had good readability and strong joint, but left a badly pitted surface.

Copper plating the granules and applying with dilute glue gave a strong bond, but with almost no control. Fillet growth was extremely rapid, resulting in poor visual appearance.

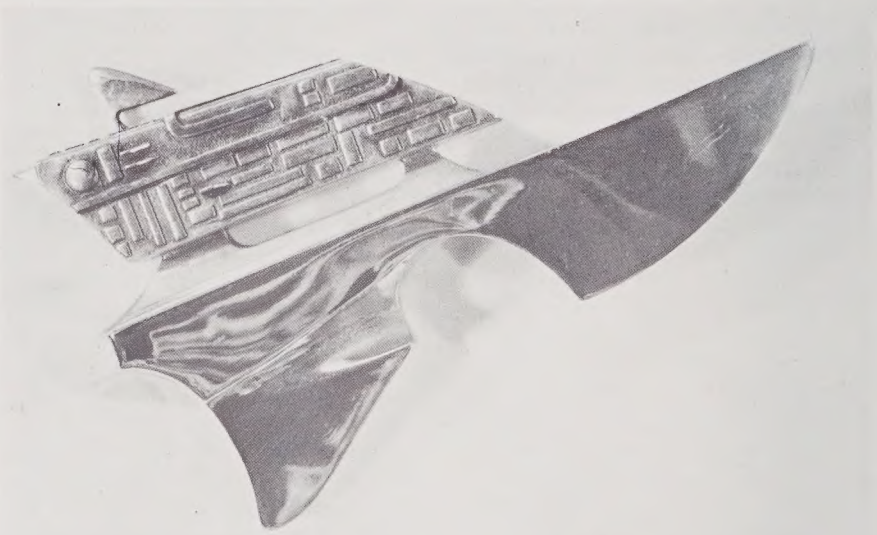
Results on most formulas improved slightly with the addition of borax glass as a flux, a suggestion by Ware (no. 11) in dealing with granulation on sterling. For a brief comparison of the best six formulas, see the comparison chart (Fig. D).

Experimentation using Ware's formulas (no. 12) on sterling was unsatisfactory, giving a poor bond with poor appearance and limited readability; in short, fusion took place very close to the melting point of sterling, leading to distortion, reticulation and total surface fusion.

Unable to reproduce Ware's results with either his formula or our own, we turned to oxidation hardening silver or Spring Silver Alloy No. 15065 (no. 13). This alloy of silver (99.65%), magnesium (.2%) and nickel (.15%) has the advantage of handling very much like fine silver while having, when heat treated, the hardness of hard-rolled sterling (no. 14) and the tensile strength of mild steel (no. 15). Every chemical formula used on oxidation hardening silver gave results which equal those on fine silver.

In addition, we found that the granulation process annealed the alloy to some extent, requiring a subsequent heat treatment in an oxidizing atmosphere for hardening.

We began work on gold using our own alloy of green gold at the suggestion of an article on granulation by Kulicke and Stark (no. 16). This bias was corroborated by the knowledge that almost all of the ancient granulated pieces are of "native gold", that is gold alloyed with up to 5% silver (no. 17).



William Ard: granulation experiment "Rocket Fetish", 3"x2" in sterling and fine silver, granulated pattern with Antimony 0_3.

11. Metal Fusion and Granulation; Ware
12. antimony 0_3 (50%), silver oxide (20%), borax (30%).
13. Oxidation Hardening Silver; Lechtzin (Goldsmiths Journal).
14. ibid.
15. Metallurgy of Non-Ferrous Metals; Dennis.
16. Workshop: Granulation; Kulicke & Stark (Craft Horizons).
17. Greek Gold; Hoffman & Davidson; p. 22.

	Relative Temperature at Fusion	Readability	Filletting	Strength	Appearance
ANTIMONY 0_3 30/1	High	Very Easy	Excellent	Excellent	Excellent
CHLORIDE-ANTIMONY 80/20	High	Easy	Excellent	Excellent	Excellent
CU CHLORIDE 110/1	Low	Very Easy	Very Good	Very Strong	Good
CU ACETATE 110/1	Medium	Good	Good	Good	Good
CU HYDRATE 30/1	Medium	Good	Good	Fair	Very Good
CHLORIDE-ZINC-TIN 30/1	Very High	Good	Good	Good	Fair

FIGURE D: Comparison chart of best formulas on silver in order of apparent reliability.

Experimentation yielded several formulas, including copper plating, which worked very well on 22½k, 16k and 14k green gold. It is expected that granulation could be done on the whole spectrum of gold to silver alloys using one or another of the formulas.

The best of the green gold formulas follow:

1. Silver Nitrate 20/1 was the best formula for 22½k green gold, but failed to work on the lower karat alloys. It gave a strong bond with excellent fillets and a bright, burnished appearance. The fusion flash was easily readable.
2. Antimony 0_3 with flux, unlike silver nitrate worked only on the lower karat golds, giving a good, strong bond and excellent surface quality. Readability was not as clear as with chloride or copper plating; fusion temperature was also higher.
3. Cu Chloride 20/1 appeared to be the most reliable chemical for green gold and fused at the lowest temperature. Fusion read as a bright, liquid "wink" leaving excellent fillets and a strong bond, as well as an excellent visual appearance.
4. Cu Hydrate 30/1 also gave a strong bond with excellent surface characteristics, but fused at a much higher temperature and was more difficult to read.
5. Copper plating and glue 5/1, with or without flux, fired slightly lower than chloride and was not as easily read, but gave excellent results in both strength and appearance. The granules were plated by pickling in an iron pan with a few grains of copper sulphate. Only a light coating was necessary; a copper residue was left on the surface after firing if the plating was too heavy.

Acetate, Zinc Hydrate, Silver Oxide and Copper Hydroxide were also experimented with and dropped due to inferior results. Cu Chloride-Zinc-Tin and Cu Chloride-Antimony 0_3 were also used with some excellent results, but the results were inconsistent due to difficult readability.

Experimentation with commercial yellow gold ran the same course. With 18k gold the lowest firing formulas and the easiest to read were the experiments with copper plated granules and glue 5/1, with or without flux. In both cases there was an obvious fusing "wink" and the joint was strong with light filletting and an excellent appearance after pickling. The use of flux notably reduced the firing temperature and kept the surface cleaner.

Silver Nitrate 20/1, Antimony 0_3 10/1 and Cu Chloride 10/1 all achieved excellent results with good readability. Each chemical produced a slightly finer fillet structure than either plating process.

Finally, 18 karat gold was granulated to itself using only dilute glue and flux. Using the copper in the alloy itself to form the bond, this produced an excellent fillet structure, strong joint and clean surface, but fired much higher than all the other methods. An attempt was made to eliminate the glue as well, but the lack of carbon pushed the fusion temperature into the sweating range.

Our experimentation with 14 karat yellow gold yielded results as dismal as that on the sterling. The comparatively low gold content made the metal unstable and none of the formulas fused well before the melting point was reached. Further experiments with other 14 karat alloys and plating processes are indicated before the alloy can be successfully granulated as Conrad Brown suggests (no. 18).

Testing in bond strength was conducted on fine silver and oxidation hardening silver to determine the value of localized fusion welding in the construction of hollow forms. The top eight chemical formulas for silver were tested against hard solder for filletting, strength and finish.

Each chemical was used to fuse a ½"x1/8" 22 guage wall to a base plate of the same metal. The test pieces were then twisted and bent to destroy the bond.

Most of the chemicals had extraordinary bond strength, the test pieces being distorted beyond recognition before failure. Unquestionably, the toughest joint was from the chloride and antimony 0_3 80/20, with very heavy fillets. Close behind were antimony 0_3 alone and chloride-antimony 0_3 50/50, which fired much cleaner with very light filletting.

The only unsatisfactory formulas of the best eight were Cu Hydrate and Chloride-Zinc-Tin, possibly through consistent underfiring. These, though fairly strong, were not as tough as the hard solder.

The drawbacks of fusion welding for silver construction may outweigh the benefits. Careful fitting is absolutely necessary as slight gaps or warpage will not fill in as with solder. However, the piece can be reheated to fusion temperature repeatedly and, without re-application of the solution, the joint will remain firm. This could be of great advantage in a complicated construction.

CONCLUSION—What began as a search in earnest to discover the mysterious process of Etruria actually turned up a great deal of written information. And, as Littledale states, we still haven't found the right way, the only way of working in granulation.

Though we are confident of the formulas listed here, we are also confident that further bracketting and experimentation could lead to success on the less hopeful formulas. We can only stress the value of practical experience. In the words of Biringuccio's Pirotechnia (no. 19) "try everything, to vary the proportions and the means, until the best way is found."

We must admit to a change of purpose in mid-project. What began with consciously innovative experimentation in the mokume process, gradually became the semi-scientific evaluation of published information on granulation with the intent to produce a comprehensive guide for studio application.

Research and experimentation into granulation will continue, however, at Southern Illinois University. A grant has been applied for to subsidize further research and we have begun to experiment with other procedures and other kinds of metallic alloys.

18. Gold Granulation; Brown (Craft Horizons).

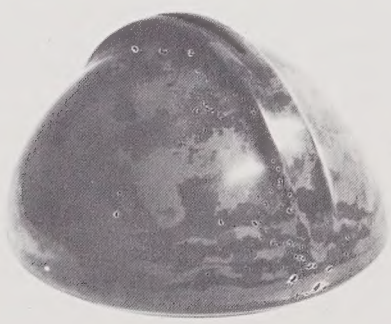
19. Mappae Clavicula; trans. Hawthorn & Smith.

Concerning our return to the forge, we used the torch for most of our experiments. A compressed air charcoal brazier was designed and built for use in the studio, but we cannot recommend it over the torch. It does provide a better reduction environment which produced a slow-motion fusion effect, at least with antimony 0_3, and it gives a more mature fillet structure by heating from underneath. But ranked against constant stoking and fire maintenance it seems a small return.

What we suggest is an exercise in comparative technologies, a consideration of the old methods if not a return to them. The Etruscan metalsmith may have reached for his charcoal brazier simply because he didn't have a torch at his bench, but there was something about the technology that created the brazier that made it easy for him to baffle us for two thousand years. The Japanese, too, had no choice about his tools, but they gave him an edge over us in his work.

As modern metalsmiths, we need to change our approach. Nothing in our art need be closed to us if we are willing to use our technology along with that of the past and not instead of it.

Marvin Jensen: Bank (copper, shakudo, shibuichi, kuromi-do) 4½"



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